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Author(s)	Sundfeld, RH; de Oliveira, CH; da Silva, AM; Briso, AL; Sundfeld, ML
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Original Article

Resin Tag Length of One-Step and Self-Etching Adhesives Bonded to Unground Enamel

Renato Herman Sundfeld, Carlos Henrique de Oliveira, Adriana Maria Jorge Dal'acqua da Silva, André Luiz Fraga Briso and Maria Lúcia Marçal Mazza Sundefeld*

Department of Restorative Dentistry, Araçatuba School of Dentistry, Paulista State University, UNESP, Rua José Bonifácio 1193, CEP: 16015 050, Araçatuba, São Paulo, Brazil

** Department of Biostatistics, Araçatuba School of Dentistry, Paulista State University, UNESP,*

Rua José Bonifácio 1193, CEP: 16015 050, Araçatuba, São Paulo, Brazil

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Abstract

Length of resin tags yielded by utilization of an one-step conventional adhesive system and self-etching adhesive system on unground enamel was observed. In study Groups I and III, the enamel surface was etched for 60 seconds with 35% phosphoric acid gel and adhesive systems PQ1 (Ultradent Products, Inc) and Adper Prompt L Pop (3M/ESPE) were applied. Adper Prompt L Pop (3M/ESPE) was also applied in Group II in accordance with the manufacturer's recommendations. After application of these adhesive systems to dental enamel, specimens were prepared for light microscopy analysis to ascertain degree of penetration ($\times 400$). The results were submitted to an analysis of variance at the 5% level; whenever there was significance, the Tukey test was applied at the 5% level. It was found that acid etching prior to application of conventional and self-etching adhesive materials provided higher penetration of the adhesive into the unground enamel surface compared to that achieved solely by application of self-etching adhesive.

Key words: Unground enamel—Conventional adhesive system—
Self-etching adhesive system—Light microscopy analysis—
Resin tags

Introduction

The bonding of adhesive materials to an acid-etched enamel structure is classically considered to be a micromechanical process, since the etchant allows the adhesives to penetrate by means of capillarity, leading to

formation of resin tags that yield a highly effective mechanical interlocking between resin and surface^{4,8,18}.

However, new materials and methods continue to be proposed for the etching of dental tissues. These have included two-bottle adhesive systems, one-bottle systems²¹, and

self-etching adhesives for both enamel and dentin, which eliminates the need for an etchant, thus allowing a procedural simplification and time to be saved^{3,5,9,12-14}.

The application of self-etching adhesives to an enamel surface remains a controversial issue, particularly with regard to their application to unground enamel. Some researchers have supported the manufacturers' suggestion that combined use of phosphoric acid etching is necessary when bonding is to be done on a substrate^{11,14}. Others, however, have found no differences in bond strength between that achieved with mild self-etching and that with total-etch adhesives when bonding to an unground enamel^{6,10,15}.

On the other hand, certain procedures such as bonding of orthodontic brackets^{1,2,27} and preparation for application of pit or fissure sealants always require bonding to an unground enamel⁷. In such cases, utilization of a more aggressive self-etching system might offer the more favourable alternative. The objective of this study was to examine, by means of light microscopy, the penetration of one-step and self-etching adhesives into unground enamel.

Materials and Methods

Thirty human teeth recently extracted from patients aged between 14 to 21 years were used in this study. The teeth were cleaned, washed, and polished with pumice and water, and then stored in distilled water at room temperature until used. The study protocol was approved by the Human Subject Review Committee of Araçatuba School of Dentistry, Paulista State University, UNESP (Araçatuba, SP, Brazil).

The teeth were randomly divided into 2 experimental groups and one control group of 10 specimens each. The operative procedures were initiated by prophylaxis with pumice and water applied with a rotary brush at low speed, followed by rinsing and drying. The enamel surface was not submitted to preparation for application of the adhesive

materials. The study groups were as follows:

GI (control group): The enamel surface was etched for 60 seconds¹⁸ with 35% phosphoric acid gel (Ultradent Products, Inc.). It was then rinsed for 15 seconds and air-dried. A one-step adhesive PQ1 (Ultradent Products, Inc.) was applied to the enamel surface under pressure with a brush, after which, the surface was gently air-dried for 5 seconds and light-cured for 20 seconds with an Ultralux Lens unit (Dabi Atlante) with an output of 450 mW/cm².

GII: No pre-etching was performed. The enamel surface was rinsed with water, air-dried and then the Adper Prompt L Pop system (3M/ESPE Dental Products) was rubbed on both enamel surfaces for 15 seconds, followed by air-drying for 5 seconds. Next, the applicator was soaked and a second coat of adhesive was applied, which was then air-dried for 5 seconds and light-cured for 10 seconds with an Ultralux Lens unit with an output of 450 mW/cm².

GIII: The procedure was the same as for GI, except that the Adper Prompt L Pop system was applied and cured as for GII.

Immediately after application of adhesive to the unground enamel surface, an approximately 2 mm thick Vitalescence composite resin (Ultradent Products, Inc.) was applied to the specimens in Group I, and Z 250 (3M/ESPE Dental Products) to the specimens in Groups II and III, followed by light curing for 40 seconds with an output of 450 mW/cm².

Each tooth was cut into 5 sections of approximately 200 μm in thickness in the bucco-lingual direction (Isomet-Buheller). Three sections were selected and further polished with aluminum oxide sandpaper with grits 80, 360 and 600 to a thickness of approximately 100 μm, which was checked with a digital caliper.

For analysis of the resin tags in the enamel, these sections were decalcified in 40% nitric acid for approximately 60 seconds, i.e., until the enamel was dissolved, leaving the adhesive with resin tags behind. They were then immersed in distilled water and once again mounted on glass and covered by a glass cover

Table 1 Application of the Tukey test; means of the control and experimental groups according to application of the adhesive material

Groups	Enamel Conditioning	N	Mean (Um)	Standard Deviation	Result of Tukey test
I (PQ1)	+	10	53.00	11.67	A
II (Adper)	-	10	3.58	2.68	B
III (Adper)	+	10	22.80	2.75	C

+ YES, - NO

* Same letters indicate no significant statistical difference between groups.

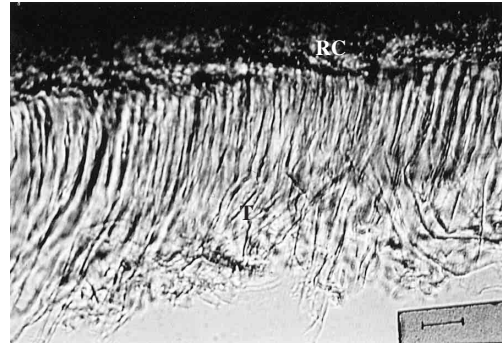


Fig. 1 Specimen representing Group I (PQ1 Adhesive) — penetration of resin tags into unground enamel, with mean length of 53.0 micrometers (Original magnification $\times 400$). RC: composite resin, T: tags.

slide. The edges were sealed with Canadian oil¹⁸⁾.

The selected sections of each tooth were analyzed and measured on a light microscope Axiophot (ZEISS DSM-940 A) at $\times 400$ magnification, with a micrometric ocular 40/075. Measurements of the resin tags of each section were performed by careful analysis of the entire extension of the ground section made by a single calibrated examiner. Three measurements were collected for each section. Consequently, for each section analyzed, the length of the resin tags corresponds to the mean of the three measurements performed. Thus, three means were obtained for each specimen. The means of the tag length corresponding to each specimen were submitted to statistical analysis using analysis of variance, with a confidence level of 5%, followed by the Tukey test.

Results

The length of the resin tags was subjected to an analysis of variance. Significant differences were found between the control (GI) and experimental groups (GII and GIII). Afterwards, the Tukey test was applied at the 5% level (Table 1).

As can be seen in Table 1, the specimens in the control group (GI), in which the PQ1

adhesive was applied, showed superior behavior with regard to penetration of adhesive material into unground enamel compared to that in the experimental groups (GII and GIII), in which self-etching adhesive Adper Prompt L Pop was applied. Application of conventional adhesive after enamel etching in the control group (GI) provided an excellent environment for the formation of resin tags, presenting longer, numerous, and uniform resin tags of similar length (Fig. 1) and contiguous with the composite resin.

Application of self-etching adhesive Adper Prompt L Pop in GII and GIII resulted in a statistically significant decrease in the penetration of adhesive material into the unground enamel. This was supported by the results of the Tukey test ($p=0.05$) (Table 1) (Figs. 2, 3 and 4). The lowest penetration of adhesive into unground enamel was recorded in the specimens of group II, in which self-etching Adper Prompt L Pop adhesive was applied in accordance with its clinical application, namely without previous acid etching. This resulted in the formation of shorter and non-uniform resin tags.

Acid etching prior to application of Adper Prompt L Pop system (Group III) allowed the achievement of longer resin tags, yet not as long as those yielded by the conventional adhesive material PQ1 (Fig. 4).

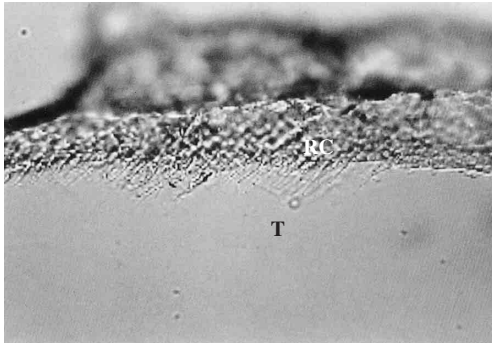


Fig. 2 Specimen representing Group II (Adper Prompt L Pop system)—penetration of resin tags into unground enamel, with mean length of 3.58 micrometers (Original magnification $\times 400$). RC: composite resin, T: tags.

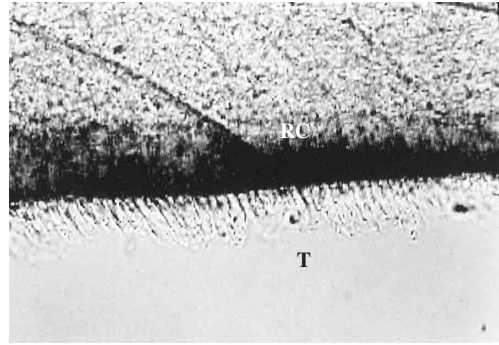


Fig. 4 Quality and penetration of resin tags in Group III (H_3PO_4 +Adper Prompt L Pop system); resin tags presented mean length of 22.80 micrometers and were much better than those in group II (only Adper Prompt L Pop). (Original magnification $\times 400$). RC: composite resin, T: tags.

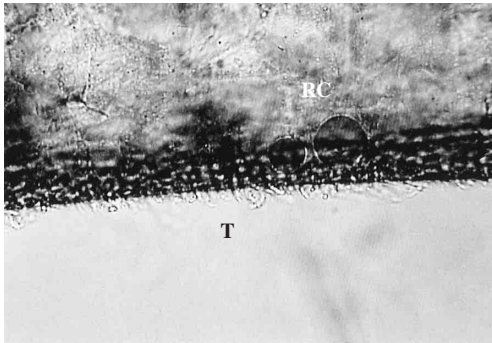


Fig. 3 Specimen representing Group II (Adper Prompt L Pop system)—penetration of resin tags into unground enamel, with mean length of 3.58 micrometers (Original magnification $\times 400$). RC: composite resin, T: tags.

Discussion

The conventional one-bottle adhesive material PQ1 consists of a combination of hydrophobic and hydrophilic components. It is designed to reduce the necessary number of procedural steps and is suitable for use with both enamel and dentin after proper etching of each structure with phosphoric acid. The present study found that application of this system allowed greater penetration of adhesive material into the dental enamel, with formation of uniform, long, and well-distributed resin tags throughout the interface between

enamel and resin of a mean length of 53.0 micrometers.

Acid etching of dental enamel prior to application of a resin material considerably influences the durability of that material in the oral cavity. One such example is the accomplishment of pit and fissure sealants reported by Simonsen in 1991¹⁷⁾ and Sundfeld *et al.* in 2004¹⁹⁾, at 15 and 11 years after application, respectively. This was mainly due to the extremely accurate technique to which the sealing material was submitted, comprising application on etched, well-dried, and non-contaminated enamel.

Self-etching adhesive materials were introduced in an attempt to further simplify the operative steps involved in achieving bonding to the surface of a tooth. Such materials are composed of acidic monomers, solvents, additives, photoinitiators and water, and thus are capable of yielding acid etching followed by adhesive penetration into the tooth structure, as stated by the manufacturers. They are generally considered to be less technique-sensitive compared to systems that utilize separate acid etching and rinsing steps. In fact, Sundfeld *et al.* in 2005²¹⁾, using a methodology proposed by Tay *et al.* in 1995²²⁾ utilized light microscopy to observe the application of self-etching adhesive Adper Prompt L Pop on an intact dentin tissue and showed that such aggressive self-etching capable of deminer-

alizing subsurface dentin and forming an authentic hybrid layer, presenting a mean thickness of around 3.13–3.72 μm and resin tags with mean lengths ranging from 13.81–19.30 μm . These adhesive interfaces were morphologically similar to those produced by conventional adhesives in the dentin tissue²⁰, as revealed by light microscopy analysis.

However, as regards the application to highly mineralized enamel structures, much has been questioned on the actual effectiveness of these new adhesive materials. Such concern is, to some extent, related to the higher calcium content of this substrate; its initial acidity; the lower acidity of its acidic monomer when compared to phosphoric acid; the impossibility of eliminating water during the bonding process, since it is part of the material; the buffering capacity of the tooth structure; and its inherent hydrophilic characteristics^{5,12,23}.

Light microscopy showed that a combination of these factors significantly contributed to the lower penetration of the self-etching adhesive material Adper Prompt L Pop into the enamel surface not submitted to etching with phosphoric acid, which presented a mean length of 3.58 micrometers on the enamel surface.

In 2004, Carvalho *et al.*⁵ reported that, because self-etching adhesives are acidic and not rinsed from the tooth surface, they are not able to yield unlimited demineralization of the tooth tissues some time after application, and that the mineralized tooth structure is able to buffer the acidic elements of the materials, thus neutralizing their demineralizing action some seconds after application. Therefore, it should be noted that such materials have the advantage of establishing their own means of penetration into the tooth substrate, and that the extended demineralization that they offer may then be completely filled by the adhesive agent, thus reducing the creation of porosities at this area, which, according to Marquezini *et al.* in 2003¹², may increase its degradation over time. Consequently, such factors may lead to limited penetration of the adhesive system, restricting

it to the more superficial enamel layers with shorter resin tags, as observed in the present study.

The first self-etching primers (two-step) were characterized by their lower acidity when compared to self-etching adhesives (one-step), which led them to be ineffective in achieving bonding to enamel⁹. On the other hand, the development of more acidic formulations such as that of the self-etching system Adper Prompt L Pop, while making them more aggressive in terms of etching ability, also made them insensitive to the characteristics of the enamel substrate. According to Van Meerbeek *et al.* in 2003²⁰, this allows them to form hybrid layers in enamel that are morphologically similar to those yielded by conventional systems. However, self-etching adhesive systems (one-step) present the lowest bond strength values both in enamel and dentin due to their structural fragility to tension, leading to cohesive rupture i.e., failure within the adhesive⁵. By extrapolation of these findings to those achieved in the present study, it may be assumed that the lower penetration of such adhesive systems into unground enamel surfaces when compared to that of conventional adhesives may to some extent lead to low tensile strength. So far, there are no conclusive and specific reports available relating the length of resin tags to their bonding ability to dental enamel for the materials examined in this study. However, Shinchi *et al.*¹⁶, in 2000, observed that the tensile bond strength (10 MPa) of a conventional light-cured resin to enamel pre-treated with various acid concentrations did not vary significantly. However, resin tag length was found to decrease significantly from 22 μm for 35% H_3PO_4 to 12 μm for 20% H_3PO_4 , to 9 μm for 5% and 10% H_3PO_4 , and to 5 μm for 3% H_3PO_4 .

For comparison, the Adper Prompt L Pop system was also applied on dental enamel previously etched with phosphoric acid (Group III), showing a penetration mean of 22.80 micrometers into the etched enamel surface, yet displaying a significantly lower penetration than with the one-bottle conventional

adhesive PQ1 (Group I) which yielded a mean penetration of 53.0 micrometers. It was still significantly higher than that observed for the enamel not etched with phosphoric acid (Group II), though, which yielded a mean penetration of 3.58 micrometers into the enamel surface. This procedure, which was similar to that for the conventional system PQ1, yielded a higher rate of infiltration.

These findings suggest that the penetration capacity (resin tags) of the self-etching adhesive system Adper Prompt L Pop might be significantly improved by previous acid etching of the dental enamel. This is clinically relevant, as retention is fundamental and dependent on the presence of dental enamel¹²⁾. Therefore, utilization of previous acid etching or removal of a prismatic enamel layers, either by grinding or by preparation of a bevel on the cavity margins^{24, 25)} may result in superior retention. Certain procedures such as bonding of orthodontic brackets and preparation for application of pit or fissure sealants²⁵⁾ always require bonding to an unground enamel. In such cases, utilization of a more aggressive self-etching system might offer the more favourable alternative. Therefore, since there are doubts on the actual efficacy of self-etching adhesive systems, investigation of the longevity of their bonding to enamel is of clinical interest in providing a better understanding of their bonding mechanisms.

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Reprint requests to:

Dr. Renato Herman Sundfeld
 Disciplina de Dentística,
 Faculdade de Odontologia
 de Araçatuba, UNESP,
 Rua José Bonifácio 1193,
 CEP: 16015 050, Araçatuba,
 São Paulo, Brazil
 Fax: 55-18-3636-3332
 E-mail: sundfeld@foa.unesp.br